## Containerless Processing of YBa2Cu3O7-d Superconductors

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The production of bulk YBa2Cu3O7–d (YBCO) and other high-Tc superconductors that maintain high-current-carrying capabilities has been hindered by weak connectivity along grain boundaries, reactions at elevated temperatures with the container materials, incongruent melting behavior, and phase instabilities.

Containerless processing provides a partial solution to these difficulties. Crucible contamination is eliminated, allowing access to the high-temperature portions of the phase diagram. In addition, containerless processing affords the possibility to deeply undercool melts leading to unique solidification paths and microstructures. This degree of undercooling should lead to enhanced physical properties, in particular, improved critical currents (Jc).

Containerless processing was performed using pure YBCO powders and small spheres in a drop tube, as well as an aero-acoustic levitation device. The drop tube (fig. 134) consists of a graphite element

**Heating Tape** Coolant Shaker Table Nichrome Booster Heater Thermocouple Zirconia Muffle Tube Power Controller Pyrometer Supply Viewport CR Gas Mixer Quartz Tube Copper Plate

FIGURE 134.—Schematic of the drop tupe.

muffle furnace, a zirconia muffle tube, oxygen atmosphere control and associated electronics. The levitation device was a single dry air jet coupled to a three-axis acoustic positioner. In this device, the small spheres were heated using a CO2 laser. Two video cameras recorded the experiments and monitored transient thermal events. During the experiment, the samples were annealed (kindled) for several minutes at 1,270 K and then heated quickly to about 2,070 K. They were then cooled rapidly (300 to 400 K/sec). A pronounced recalescence (increase of heat) was observed in some samples upon cooling. After solidification, those spheres that showed pronounced recalescence were annealed in oxygen at 1,210 K for 14 hr and then cooled slowly to 690 K and held for 40 hr to induce the tetragonal-to-orthorhombic transformation and the superconducting state. SQUID magnetometer measurements were performed to calculate intragranular critical current densities. X-ray diffraction and microstructural analysis were also carried out on preannealed and postannealed samples. The microstructures developed in the samples were primarily single-phase tetragonal YBCO and YBCO with several other related phases. In both cases, a dendritic structure appears in clusters. The formation of tetragonal YBCO indicates that the liquid was undercooled to below the peritectic temperature for this phase. Upon annealing in oxygen, the samples became single-phase orthorhombic YBCO with randomly oriented grains of 10 to 20 mm in size. The outer surface of the sample shows a 70-mm layer with columnar structure. The magnetic data indicate an intragranular Jc of  $4 \times 103 \text{ A/cm}^2$  at 50 KOe and 5 K, using the Bean critical state model (fig. 135). If the actual grain size is used (at 20 mm) in the calculation, the Jc values would be two orders of magnitude higher. The samples processed in the drop tube show similar behavior, and structural and physical properties. Both the structure and properties of processed YBCO depend on the degree of undercooling, and deeply undercooled samples show excellent critical current densities in high magnetic fields. Containerless processing of oxide supercon-

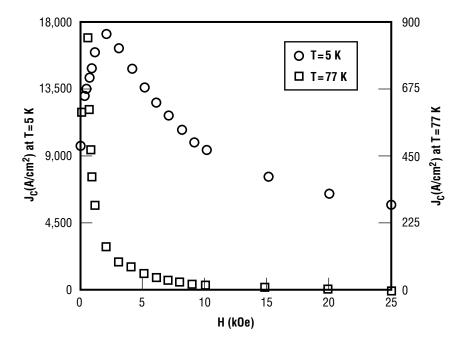


FIGURE 135.—Magnetic intragranular critical current density versus applied magnetic field at 77K and 5K.

ductors can lead therefore to deep undercooling prior to solidification and result in enhanced superconducting properties.

Associated work was also carried out in optimizing the processing parameters of pure phase YBCO and Bi-based superconductors. The preheat treatment appears to be a very important parameter in achieving this objective. The synthesis of pure phases in the Bi-based system also involves effects due to oxygen partial pressure, time and temperature. The optimization of this processing is a key step toward the successful continuation of superconducting materials development and eventual application. In conclusion, the objectives of processing oxide superconductors by containerless methods and optimizing their preparation have been attained and have led to enhanced Jc under magnetic fields. The significant improvement in Jc would result in a major step toward the use of such materials in the Materials Processing in Space Program and in other NASA-specific ground and space applications such as

shielding, magnetic levitation and magnetic bearing development.

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**University Involvement:** Vanderbilt University

Biographical Sketch: Dr. Vlasse is a materials scientist in the Microgravity Sciences and Applications Division at MSFC's Space Sciences Laboratory. He conducts research in the area of oxide superconductors and their applications, as well as in crystal growth of organic and macromolecular materials. Vlasse earned a Ph.D. in crystallography from the University of Pittsburgh and a D.Sc. degree in materials science from the University of Bordeaux.